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THE STRUCTURE AND CERTAIN PROPERTIES  
OF GAS-FLAME COATINGS

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- USSR -

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THE STRUCTURE AND CERTAIN PROPERTIES  
OF GAS-FLAME COATINGS

- USSR -

[Following is the translation of an article by K. S. Shlyakova in the Russian language publication Trudy Instituta Metallurgii imeni A. A. Baykova -- Metallurgiya, Metallovedeniye, Fiziko-Khimicheskiye Metody Issledovaniya (Works of the Institute of Metallurgy imeni A. A. Baykov -- Metallurgy, Metallurgical Science, Methods of Physico-Chemical Investigation), Moscow, No 10, 1962, pages 194-201.]

Protective coatings against corrosion and oxidation at high temperatures can be obtained by various methods, such as diffusion, saturation, electrodeposition and metallization.

Metallization is one of the advanced methods used for obtaining protection from oxidation at high temperatures, especially at short exposures to heat.

In this study, which was made in 1957, gas fired spray coatings were used. The aims of the experimentation were to determine:

- 1) the influence of heat treatment on the microstructure of the coating and on its microhardness;
- 2) the role of undercoating in achieving cohesion between the coating and the base;
- 3) the effects of vacuum treatment on the quality of the spray coatings;
- 4) comparative data on heat resistance.

The specimens were made of steel #3 and chromium-nickel steel in the form of small cylinders, 12 mm in diameter, 25 mm long and tablets 25 mm long, 12 mm wide, and 3 mm thick. Before spray coating, the specimens were sand blasted in order to obtain a rough surface for securing a better cohesion between the coating and the base. For the coating we used nichrome, alloy #2 and aluminum oxide. Nichrome and alloy #2 were used in the form of wire from two to three millimeters in diameter, and the aluminum oxide in the form of die pressed rods three millimeters in diameter.

After receiving the spray coating, the specimens were enclosed in welded quartz ampoules, heat treated in a silit oven at temperatures of 900, 1000, 1100 and 1200 degrees centigrade for one to five hours and cooled at air temperature. Experiments on heat resistance were made in a silit over at 1100 degrees for 100 hours with interruptions every 20 hours for weighing and inspection of the specimens. The heat resistance was measured by the increase in weight of the specimens expressed in grams per square meter per hour.

The Effect of the Heat Treatment on the Microstructure  
and Microhardness of the Coating

The metallization coatings are of a complex structure and are composed of a large number of small flat particles irregularly superimposed one on the other. ([Note:] The cohesion between particles of the coating is determined chiefly by the molecule adhesion between the liquid and solid particles and by the action of these forces after the solidification of the liquid metal).

Between layers of the coating, interlayers of oxides are formed. The distribution of oxides in the coating is irregular (see figure 1).

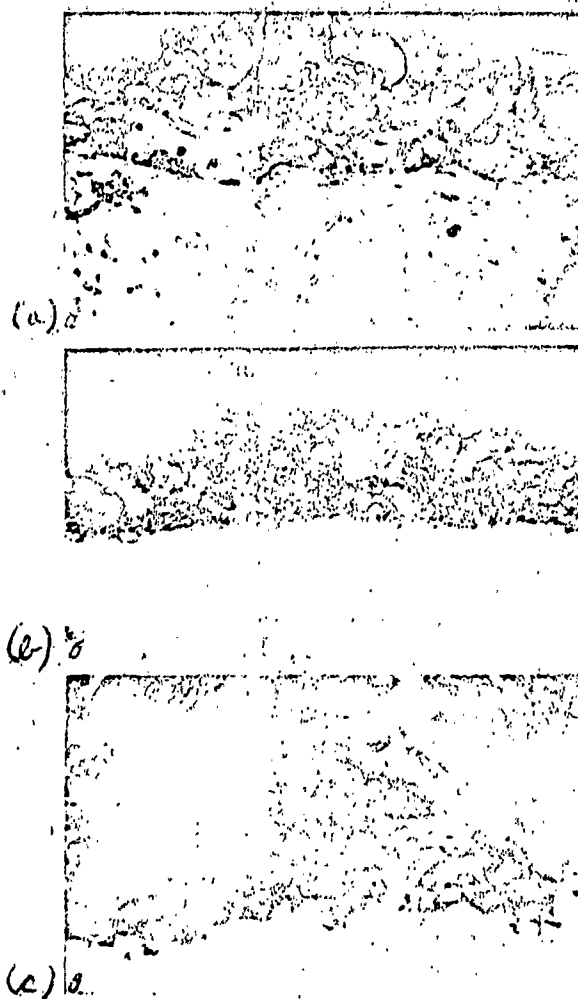


Figure 1. Microstructures of specimens of chromium-nickel steel after coating with a- nichrome 80/20; b- alloy #2; c-  $Al_2O_3$ .

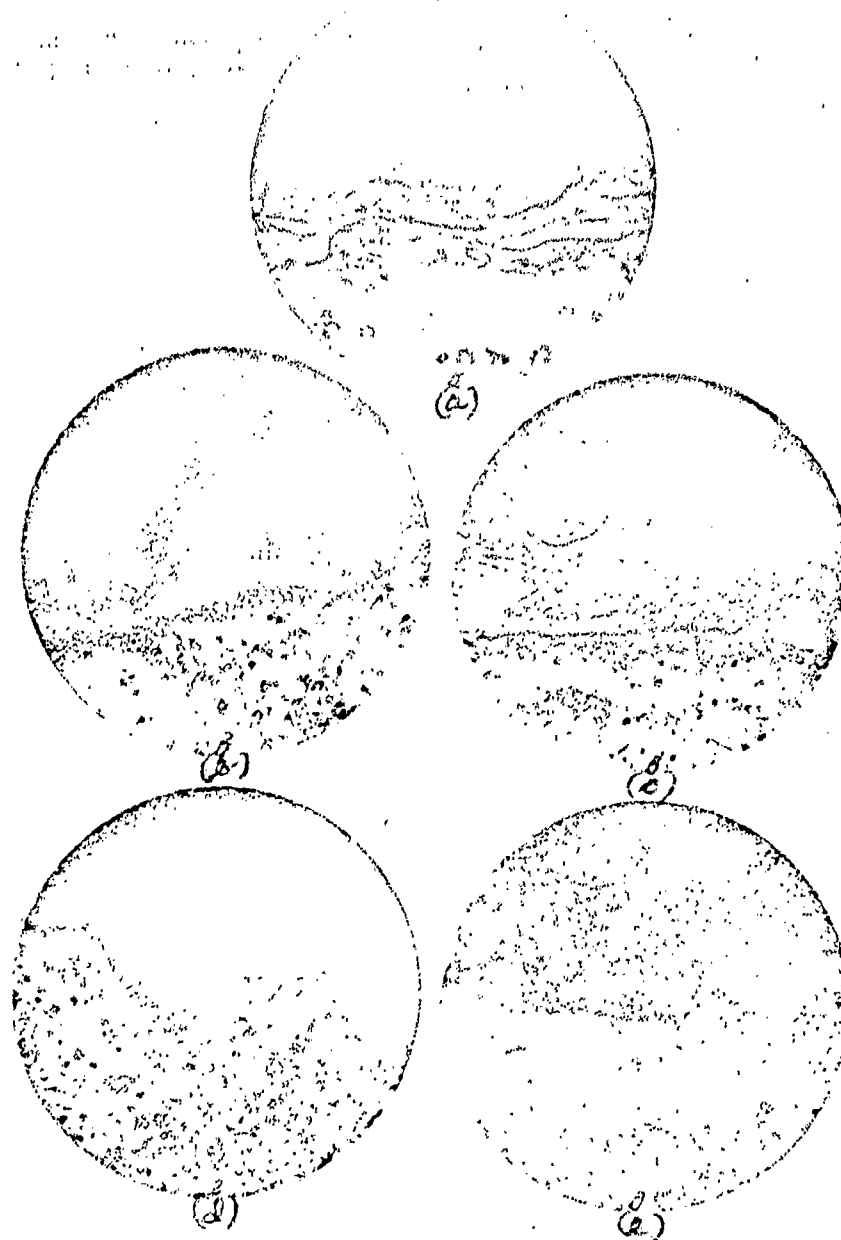


Figure 2. Microstructures of specimens of chromium-nickel steel with a nichrome spray coating 80/20. Mag. 200 X.

- a - after the coating;
- b - after heat treatment at 1000°C for one hour;
- c - heat treatment at 1000°C for five hours;
- d - heat treatment at 1100°C for one hour;
- e - heat treatment at 1100°C for five hours.

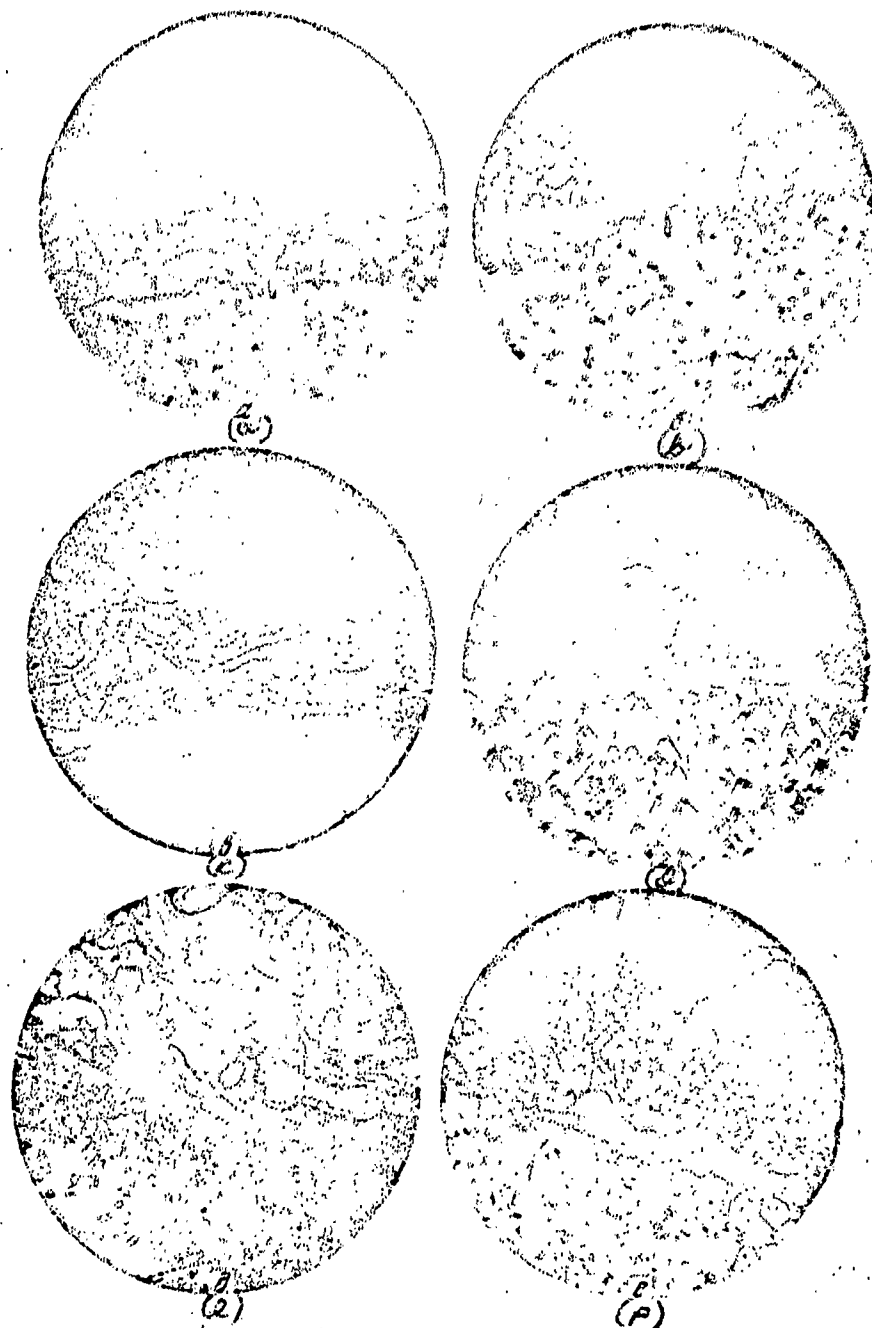


Figure 3. Microstructures of specimens with base of chromium-nickel steel and coating of alloy #2. Mag. 200 X.

a - after spray coating; b - heat treatment at 900° for one hour; c - heat treatment at 900° for five hours; d - heat treatment at 1000° for one hour; e - heat treatment at 1000° for five hours; f - heat treatment at 1100° for one hour.



Our experiments have shown that the density of the metallization coating is increased through subsequent reheating. The coating changes from porous to compact and the hardness increases correspondingly. The effect of a subsequent reheating on the microstructure at the border layer differs depending on the composition of the coating: in the case of a nichrome coating, a subsequent reheating will produce a diffusion zone at the boundary between the base metal and the coating; in the case of a #2 alloy coating, the reheating will result in the formation of an intermediate layer (see figures 2 and 3). The formation of a diffusion zone or an intermediate layer between the coating and the base does not always act favorably on the strength of the cohesion between the coating and the base. The alloy, or any other combination formed as the result of the diffusion in accordance with the diagram of these materials, is undergoing changes with temperature variations and with the length of time.

#### The Effect of An Undercoating On the Quality of Cohesion

It is known that besides the composition of the coating, many other factors influence the resistance to oxidation, namely: the preparation of the surface, the chemical composition of the base, the cohesion between the base and the coating. The latter, in its turn, depends on a number of factors. The role of the undercoating is to provide a good cohesion with the base, and that of the principal coating is to impart to the piece characteristics required for its use.

In our experiments we used aluminum oxide for the principal coating because of its high melting point and good corrosion resistance. However, aluminum oxide has a poor surface wetting ability. This was shown in our experiments and was also mentioned in other publications (see bibliographic reference 2).

In order to improve the cohesion between the aluminum oxide coating and the base metal we tried out a nichrome undercoating.

It is known that chromium steels have a high corrosion resistance due to the formation of a  $\text{Cr}_2\text{O}_3$  oxide film on the surface. In the same way in our case, a  $\text{Cr}_2\text{O}_3$  oxide film is formed on the surface of the nichrome undercoating. It is easily soluble in aluminum oxide and secures therefore a strong cohesion of the aluminum oxide coating with the base.

Beside promoting cohesion, this undercoating serves as a barrier impeding the diffusion of the base metal in the coating.

#### Heat Resistance

As a general rule, metals used in the industry include gases such as hydrogen and nitrogen. We checked the effect of vacuum treatment, resulting in elimination of gases, on the heat resistance of the coatings. Specimens of steel #3 coated with aluminum oxide and with a nichrome undercoating were placed in a vacuum over type TVV-2 and heated to  $1200^\circ$  for two hours (vacuum  $10^{-3}$ ). The specimens were then checked for oxidation. On figure 4 are shown microstructures of specimens without

and with vacuum treatment and also their microhardnesses; on figure 5 -- the appearance of the specimens after a heat resistance test; figure 6 -- demonstrates the curves of heat resistance of the various coatings used in the experiments.

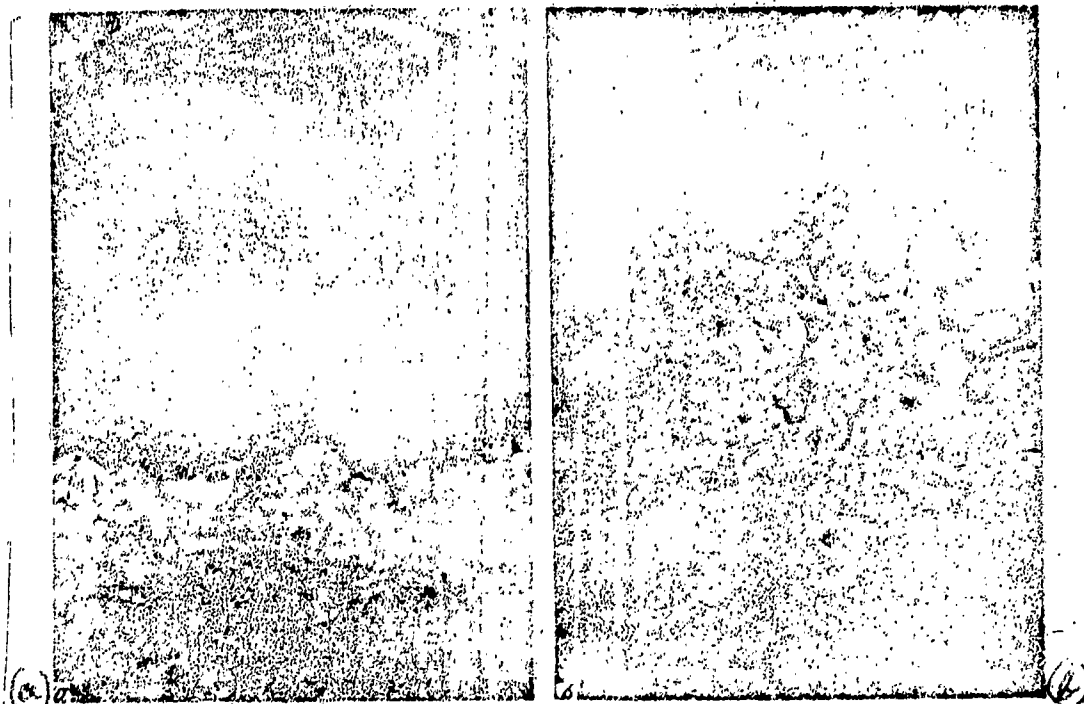


Figure 4. Microstructure of specimens (a) after spray coating and (b) after vacuum treatment at 1200° for two hours. Mag. 200 X.

As it can be seen from these curves, the nichrome coating increases the oxidation resistance of steel #3 approximately 12 times. The  $Al_2O_3$  coating with a nichrome undercoating increases it approximately 30 times. The vacuum treatment still further improves the protective quality of the coatings.

The high resistance to oxidation of vacuum treated specimens of steel #3 coated with  $Al_2O_3$  can probably be explained, first, by the selective evaporation of chromium and aluminum and, second, by the decrease of hydrogen content in the base and in the coating with the resulting decrease of stresses at the boundary between the base and the coatings. As a result, the quality of cohesion is improved and, therefore, the protective efficiency of the coating is increased.

The appearance of the vacuum treated specimens did not change after a heat resistance test at 1100° during 100 hours. The edges, which are the weakest spot of the coatings, were not destroyed by oxidation.

The surface of those specimens which were not submitted to vacuum treatment did not always remain in satisfactory condition (see Fig. 5).

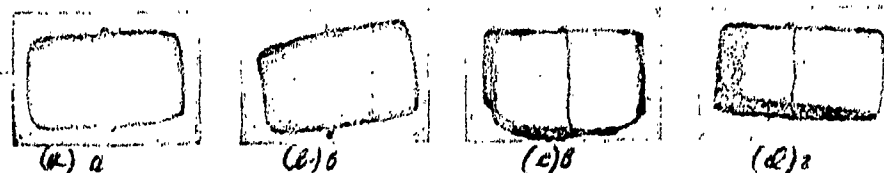


Figure 5. Specimens of steel #3 with different coatings, after heat resistance test at  $1100^{\circ}$ : a - without spray coating, after 80 hours; b - NiCr coating, after 100 hours; c - NiCr +  $Al_2O_3$  coating, after 100 hours; d - NiCr +  $Al_2O_3$  coating and vacuum treatment, after 100 hours.

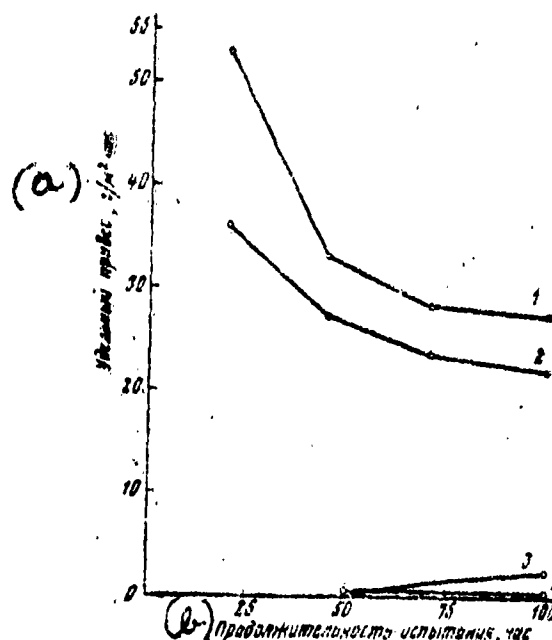


Figure 6. Heat resistance of specimens of steel #3 with various gas-flame spray coatings: 1 - NiCr; 2 - NiCr; 3 - NiCr +  $Al_2O_3$ ; 4 - NiCr +  $Al_2O_3$ , vacuum treated. Temperature  $1100^{\circ}C$ .

Legend: (a) Specific increase of weight in grams/square meters per hour.  
(b) Time; hours.

#### X-rays Testing of the Structure

Figure 7 shows radiograms of the surface of two specimens spray coated with aluminum oxide with a nichrome undercoating, after a heat resistance test at  $1100^{\circ}$  for 100 hours. One of the specimens was vacuum treated, the other was not.

The two diagrams are different. Their interpretation presents difficulties in determining whether the compound is  $\text{FeCr}_2\text{O}_3$ ,  $\text{NiCr}_2\text{O}_3$ ,  $\text{NiAl}_2\text{O}_3$ , or  $\text{FeAl}_2\text{O}_3$ . However, it can be said that the resulting compound is characteristic for a lattice of the spinel type.

In order to determine the effect of the vacuum treatment on the formation of a stable oxide film, the surfaces of the two specimens (one without vacuum treatment and the other with such treatment) were chemically analysed.

#### Chemical Analysis of the Coatings

For chemical analysis specimens of coated steel #3 with an underlying nichrome coating were dissolved in a 50 percent solution and the precipitate was analysed. The chemical composition is given in the table. The composition of the solution is not given.

As it can be seen from the table, the vacuum treated specimens contain one and a half times more Cr, three times more Al and four times less Fe than the specimens which were not vacuum treated. No nickel was found.

<u>Coating</u>	<u>State of the Specimens</u>	<u>Chemical composition, in percent</u>			
		<u>Cr</u>	<u>Al</u>	<u>Ni</u>	<u>Fe</u>
Undercoating NiCr, main coating $\text{Al}_2\text{O}_3$	After heat resistance test at $1100^\circ$ , 100 hours, air cooled.	3.21	17.14	none	44.45
Undercoating NiCr, main coating $\text{Al}_2\text{O}_3$	Vacuum treated with a subsequent heat resistance test at $1100^\circ$ , 100 hours, air cooled.	5.35	48.88	none	11.70

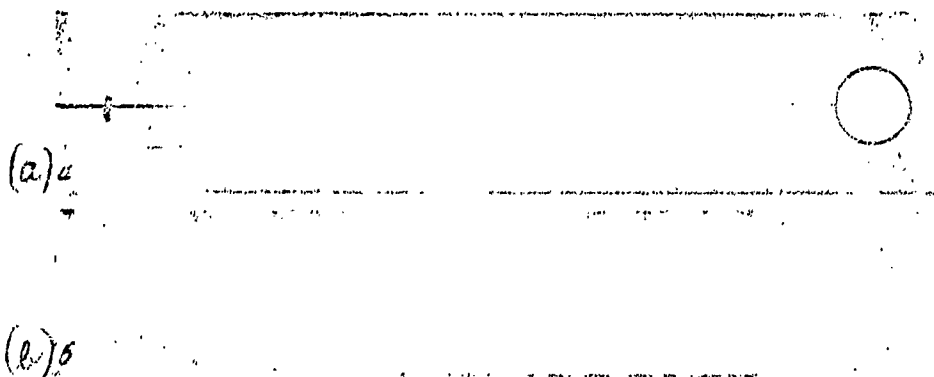


Figure 7. X-rays taken on the surface of the specimens: (a) - spray coated with NiCr +  $\text{Al}_2\text{O}_3$ ; (b) - after subsequent vacuum treatment.

This study of the chemical analyses of the coatings, of the X-rays tests, the heat resistance tests as well as of the microstructure, brings us to the conclusion that as a result of the selective evaporation of chromium and aluminum during the vacuum treatment, and of the selective oxidation during the heat treatment, a compact oxide film is formed on the surface. This film, complex in its composition, impedes the process of diffusion and thus provides a strong defensive property to the surface.

### Conclusions

1. Metallization is a technically simple method of obtaining coatings of various compositions, simple or complex;
2. Subsequent heat treatment increases the density of the coating.
3. Vacuum treatment increases the oxidation resistance of the coating.

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2. K. P. Azarov, "Wetting of Solid Phases by Melted Silicates", Doklady Akademii Nauk SSSR (Reports of the Academy of Sciences USSR), new series, 1952, 77, No 2.